UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION V

DATE: 6/17/92

SUBJECT: Review of Region V CLP Data
Received for Review on June 4. 1992

FROM: Charles T. Elly, Director(5SCRL) Patrick J. Churilla for
Central Regional Laboratory

TO: Data User: PRC

We have reviewed the data for the following case(s).

SITE NAME: Saltnalley (OH) SMO Case No. 19026

EPA Data Set No. _____ No. of Samples 12 DU/Activity Numbers 1

CRL No. _____

SMO Traffic No. EQK 13-19, EQK 30-34

CLP Laboratory: Clayton Hrs. for Review 21

Following are our findings:

See Attached Review - PJC

( ) Data are acceptable for use.
(✓) Data are acceptable for use with qualifications.
( ) Data are preliminary - pending verification by laboratory.
( ) Data are unacceptable.

cc: Elenor McLean, Sample Mgmt. Office
Edward Kantor, EMSL-Las Vegas

DATA QUALIFIERS PAGE 2 OF 10

CONTRACTOR: Clayton CASE 19026

Below is a summary of the out-of-control audits and the possible effect on the data for this case:

This review covers twelve samples, seven of which (EQK13 through EQK19) are soils and five of which (EQK30 through EQK34) are waters, for complete organic analysis at low levels except for samples EQK13 through EQK19 and EQK34, which were analyzed for volatiles only.

The reviewer's narrative and data qualifiers follow.

Reviewed by: Al Venuto (Lockheed/ESAT)

Date: 15 June 1992 Hrs. Required for Review: 18

1. Holding Times:

All samples were promptly analyzed for volatiles and easily met the fourteen day holding time from date of sampling for soils and preserved water samples.

All water samples thus analyzed were extracted for both semi-volatiles and pesticides/PCBs well within the seven day holding times for these fractions; all extracts were promptly analyzed.

2. GC/MS Tuning and GC Instrument Performance:

The GC tuning and mass calibration were all within the required Q.C. limits. All pesticide breakdown results were well below the maximum permissible limits.

3. Calibration:

The calibration outliers for all fractions are listed on the outliers forms. All RPDs in the pesticide calibration verification summaries (Pest-1) were generally well below the maximum permissible 25%. For the pesticide florisil cartridge check, the recovery of tetrachloro-m-xylene was slightly below the lower limit; since no pesticide TCL compounds were found in any of the unspiked samples, no action is recommended.

4. Method Blanks:

The water volatile method blank VBLKEAw was found to contain only the common contaminants methylene chloride and acetone; all of its associated samples also contained these two compounds except for EQK32MS and EQK33, which contained no acetone. VBLKEBw contained only methylene chloride, which was also found in both of its associated samples. The soil volatile method blank VBLKEAs contained only methylene chloride, which was also found in both of its associated samples. VBLKEBs contained methylene chloride and acetone, both of which were also found in all of its associated samples except for EQK15, which contained no acetone.

The semi-volatile method blank contained no TCL compounds but did contain two unknown TICs; all of its associated samples except EQK31 also contained the earlier eluting TIC, but only EQK33 also contained the later eluting TIC.

The pesticide method blank contained no TIC compounds.

5. Surrogate Recoveries:

All surrogate recoveries for all fractions were within the Q.C. limits.

Al Venuto
15 June 1992

6. Matrix Spikes and Matrix Spike Duplicates:

All volatile MS and MSD recoveries and RPDs were within the Q.C. limits except for the RPD for toluene in EQK32, which was above the limit; since toluene was not found in the unspiked sample EQK32, no action is recommended.

For the semi-volatile fraction, all MS and MSD recoveries and RPDs were within the Q.C. limits except for the recoveries of 4-nitrophenol in both EQK32MS and EQK32MSD, which were somewhat above the limit; since this compound was not found in the unspiked sample, no action is recommended.

For the pesticide fraction, all spike recoveries in EQK32MS were above the upper limits and the RPDs for Lindane, Heptachlor and Dieldrin were above the limits; since none of these was present in the unspiked sample, no action is recommended.

7. Field Duplicates and Field Blanks:

Sample EQK31 was identified as a duplicate of EQK30; the analyses of both were nearly identical except for a few disparate semi-volatile TICs.

Sample EQK33 was identified as a field blank; it contained no compounds other than those also found in the associated method blanks. Sample EQK34 was identified as a trip blank and analyzed for volatiles only; in addition to methylene chloride (which was also found in the associated method blank) it contained the TCL compounds acetone and chloroform.

8. Internal Standards Performance:

All volatile IS areas were within the Q.C. limits and all semi-volatile IS areas were well within the Q.C. limits.

9. Compound Identification:

All compound identifications appear to be satisfactory.

10. Compound Quantitation and Reported Detection Limits:

The correct limits were used and the proper adjustments were made for percent moisture (soil samples).

11. System Performance:

All aspects of the system performance appear to be satisfactory except that in the volatile fraction, a large peak was noted at the very beginning of each chromatogram. The results for chloromethane and vinyl chloride in each sample are therefore compromised and should be considered UJ, estimated quantiation limits, since neither was found in any sample.

12. Additional Case-Specific Problems:

None noted.

Al Venuto (Lockheed/ESAT)
15 June 1992

CALIBRATION OUTLIERS
VOLATILE TCL COMPOUNDS

CASE\SAS#: 19026 CONTRACTOR: CLAYTON

Instrument# HP-5E		Initial Cal.			Contin. Cal.			Contin. Cal.			Contin. Cal.			Contin. Cal.		
Date/Time: 4-23-92		4-23-92 10:58			5-6-92 17:26			5-7-92 12:25								
	#	rf	%rsd	*	rf	%d	*	rf	%d	*	rf	%d	*	rf	%d	*
Chloromethane	0.01															
Bromomethane	0.10															
Vinyl chloride	0.10															
Chloroethane	0.01															
Methylene chloride	0.01															
Acetone	0.01	.447	34.9	J	.556			.577	29.1	J						
Carbon disulfide	0.01															
1,1-Dichloroethene	0.10															
1,1-Dichloroethane	0.20															
1,2-Dichloroethene (total)		2.37			1.32	44.4	J	1.15	50.3	J						
Chloroform	0.20															
1,2-Dichloroethane	0.10															
2-Butanone	0.01	.630			.791	25.6	J	.635								
1,1,1-Trichloroethane	0.10															
Carbon tetrachloride	0.10															
Bromodichloromethane	0.20															
1,2-Dichloropropane																
cis-1,3-Dichloropropene	0.20															
Trichloroethene	0.30															
Dibromochloromethane	0.10															
1,1,2-Trichloroethane	0.10															
Benzene	0.50															
tran-1,3-Dichloropropene	0.10	.376			.405			.239	36.4	J						
Bromoform	0.10															
4-Methyl-2-pentanone	0.01															
2-Hexanone	0.01															
Tetrachloroethene	0.20															
1,1,2,2-Tetrachloroethane	0.50															
Toluene	0.40															
Chlorobenzene	0.50															
Ethylbenzene	0.10															
Styrene	0.30															
Xylene (total)	0.30															
Toluene-d8																
Bromofluorobenzene																
1,2-Dichloroethane-d4																

Samples affected:

	VBLK EA's	VBLK EB's
	EQK13	EQK14
	EQK16	EQK14MS
		EQK14MSD
		EQK15
		EQK17
		EQK18
		EQK19

Reviewer's Init/Date: AV 6-12-92

* These flags should be applied to the analytes on the sample data sheets.
# Minimum Relative Response Factor

3/90

CALIBRATION OUTLIERS
VOLATILE TCL COMPOUNDS

CASE\SAS#: 19026 CONTRACTOR: CLAYTON

Instrument# HP.5E		Initial Cal.			Contin. Cal.			Contin. Cal.			Contin. Cal.			Contin. Cal.		
Date/Time: 4-27-92		4-27-92 14:00			5-5-92 7:03			5-6-92 00:15								
	#	rf	%rsd	*	rf	%d	*	rf	%d	*	rf	%d	*	rf	%d	*
Chloromethane	0.01	.356			.357			.191	46.4	J						
Bromomethane	0.10															
Vinyl chloride	0.10	.526			.553			.353	32.9	J						
Chloroethane	0.01															
Methylene chloride	0.01															
Acetone	0.01	.256	37.4	J	.274			.201	29.7	J						
Carbon disulfide	0.01															
1,1-Dichloroethene	0.10															
1,1-Dichloroethane	0.20															
1,2-Dichloroethene (total)		2.80			2.90			1.44	48.7	J						
Chloroform	0.20															
1,2-Dichloroethane	0.10															
2-Butanone	0.01															
1,1,1-Trichloroethane	0.10															
Carbon tetrachloride	0.10															
Bromodichloromethane	0.20															
1,2-Dichloropropane																
cis-1,3-Dichloropropene	0.20															
Trichloroethene	0.30															
Dibromochloromethane	0.10															
1,1,2-Trichloroethane	0.10															
Benzene	0.50															
trans-1,3-Dichloropropene	0.10	.412			.419			.592	43.7	J						
Bromoform	0.10	.546			.754	35.1	J	.763	39.7	J						
4-Methyl-2-pentanone	0.01															
2-Hexanone	0.01															
Tetrachloroethene	0.20															
1,1,2,2-Tetrachloroethane	0.50															
Toluene	0.40															
Chlorobenzene	0.50															
Ethylbenzene	0.10															
Styrene	0.30															
Xylene (total)	0.30															
Toluene-d8																
Bromofluorobenzene																
1,2-Dichloroethane-d4																

Samples affected:

VBLKEA.W	VBLKEB.W
EQK31	EQK30
EQK32	EQK34
EQK32MS	
EQK32MSD	
EQK33	

Reviewer's Init/Date: AV 6-12-92

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# Minimum Relative Response Factor

3/90

W

CALIBRATION OUTLIER
SEMVOLATILE TCL COMPOUNDS
(Page 1)

CASE\SAS#: 19026 CONTRACTOR: CLAYTON

Instrument# HP-6F		Initial Cal.			Contin. Cal.			Contin. Cal.			Contin. Cal.			Contin. Cal.		
Date/Time: 5-18-92		5-18-92 12:46			5-22-92 00:43			5-22-92 19:07			5-27-92 3:07					
	#	rf	%rsd	*	rf	%d	*	rf	%d	*	rf	%d	*	rf	%d	*
Phenol	0.80															
bis(chloroethyl) Ether	0.70															
2-Chlorophenol	0.70															
1,3-Dichlorobenzene																
1,4-Dichlorobenzene																
1,2-Dichlorobenzene																
2-Methylphenol	0.70															
2,2'-Oxybis(1-chl-propane)	0.01															
4-Methylphenol	0.60															
N-nitroso-di-n-propylamine	0.50															
Hexachloroethane	0.30															
Nitrobenzene	0.20															
Isophorone	0.40															
2-Nitrophenol	0.10															
2,4-Dimethylphenol	0.20															
bis-(2-chloroethoxyl)methane	0.30															
2,4-Dichlorophenol	0.20															
1,2,4-Trichlorobenzene	0.20															
Naphthalene	0.70															
4-Chloroaniline	0.01	.196	37.4	J	.072	63.3	J	.113	42.4	J	.153					
Hexachlorobutadiene	0.01															
4-Chloro-3-methylphenol	0.20															
2-Methylnaphthalene	0.40															
Hexachlorocyclopentadiene	0.01															
2,4,6-Trichlorophenol	0.20															
2,4,5-Trichlorophenol	0.20															
2-Chloronaphthalene	0.80															
2-Nitroaniline	0.01	.501			.368	26.6	J	.462			.440					
Dimethyl phthlate	0.01															
Acenaphthylene	1.30															
2,6-Dinitrotoluene	0.20															
3-Nitroaniline	0.01	.030	32.6	J/R	.019	36.7	J/R	.074	146.7	J	.014	53.3	J/R			
Acenaphthene	0.30															
2,4-Dinitrophenol	0.01	.231			.173	25.1	J	.178			.117	49.4	J			
4-Nitrophenol	0.01	.177			.169	38.4	J	.095	46.3	J	.085	52.0	J			
Dibenzofuran	0.80															
2,4-Dinitrotoluene	0.20															

Affected samples:

EQK30	EQK32MSD	SBLK W1
EQK31		EQK33
EQK32		
EQK32MS		

Reviewer's Init/Date: AV 6-12-92

* These flags should be applied to the analytes on the sample data sheets.
# Minimum Relative Response Factor

CALIBRATION OUTLIER
SEMVOLATILE TCL COMPOUNDS
(Page 2)

CASE\SAS#: 19026 CONTRACTOR: CLAYTON

Instrument# HP-6F		Initial Cal.			Contin. Cal.			Contin. Cal.			Contin. Cal.			Contin. Cal.		
Date/Time: 5-18-92		5-18-92 12:46			5-22-92 00:43			5-22-92 19:07			5-27-92 3:07					
	#	rf	%rsd	*	rf	%d	*	rf	%d	*	rf	%d	*	rf	%d	*
Diethylphthalate	0.01															
4-Chlorophenyl-phenylether	0.40															
Fluorene	0.90															
4-Nitroaniline	0.01	.072			.061			.101	40.3	J	.045	33.3	J/R			
4,6-Dinitro-2-methylphenol	0.01															
N-nitrosodiphenylamine	0.01	.387			.256	33.5	J	.290	25.1	J	.464					
4-Bromophenyl-phenylether	0.10															
Hexachlorobenzene	0.10	.291			.370	27.2	J	.345			.328					
Pentachlorophenol	0.05	.174			.192			.177			.122	24.1	J			
Phenanthrene	0.70															
Anthracene	0.70															
Carbazole			38.8	J												
Di-n-butylphthalate	0.01	1.41			1.66			1.55			1.76	25.2	J			
Fluoranthene	0.60															
Pyrene	0.60	1.66			1.77			2.17	31.2	J	2.28	37.4	J			
Butylbenzylphthalate	0.01	.919			.972			1.16	26.0	J	1.20	30.0	J			
3,3'-Dichlorobenzidine	0.01	.106			.057	46.2	J	.023	75.3	J/R	.041	61.3	J/R			
Benzo(a)anthracene	0.80															
Chrysene	0.70															
bis(2-Ethylhexyl)phthalate	0.01	1.27			1.46			1.70	33.6	J	1.56					
Di-n-octyl phthalate	0.01															
Benzo(b)fluoranthene	0.70															
Benzo(k)fluoranthene	0.70	1.02			1.23			1.36	33.5	J	1.35	32.5	J			
Benzo(a)pyrene	0.70															
Indeno(1,2,3-cd)pyrene	0.50															
Dibenz(a,h)anthrancene	0.40															
Benzo(g,h,i)perylene	0.50															
Nitrobenzene-d5	0.01															
2-Fluorobiphenyl	0.70															
Terphenyl-d14	0.50															
Phenol-d5	0.80															
2-Fluorophenol	0.60															
2,4,6-Tribromophenol	0.01															
2-Chlorophenol-d4																
1,2-Dichlorobenzene-d4																

Reviewer's Init/Date: AV 6-12-92

* These flags should be applied to the analytes on the sample data sheets.
# Minimum Relative Response Factor

CALIBRATION OUTLIERS
PEST/PCB TCL COMPOUNDS

CASE\SAS#: 19026 CONTRACTOR: CLAYTON

Column: DB-608

Instrument#: D082	Init. Cal.		Cont. Cal.		Cont. Cal.		Cont.Cal	
Date/Time 5-16-92	5-16 42 17:47		5-21-92 14:57					
	%RSD	*	%RPD	*	%RPD	*	%RPD	*
Alpha-BHC	21.3	J						
Beta-BHC								
Delta-BHC								
Gamma-BHC								
Heptachlor								
Aldrin								
Heptachlor epoxide								
Endosulfan I								
Dieldrin								
4,4'-DDE								
Endrin								
Endosulfan II								
4,4'-DDD								
Endosulfan sulfate								
4,4'-DDT								
Methoxychlor								
Endrin ketone								
Endrin aldehyde								
Alpha chlordane								
Gamma chlordane								
Aroclor-1016								
Aroclor-1221								
Aroclor-1232								
Aroclor-1242								
Aroclor-1248								
Aroclor-1254								
Aroclor-1260								
Toxaphene								

Affected samples:	
	PBLKW1
	EQK30
	EQK31
	EQK32
	EQK32MS
	EQK32MSD
	EQK33

Pest/PCB

Reviewer's Initial/Date AV 6-12-92

3/90 Rev

* These flags should be applied to the analytes on the Sample Data Sheets.

CALIBRATION OUTLIERS
PEST/PCB TCL COMPOUNDS

CASE\SAS#: 19026 **CONTRACTOR:** CLAYTON

Column: DB-1701

Instrument#: D081	Init. Cal.		Cont. Cal.		Cont. Cal.		Cont.Cal	
Date/Time 5-16-92	5-16-92 17:47		5-21-92 14:57					
	%RSD	*	%RPD	*	%RPD	*	%RPD	*
Alpha-BHC	21.3	J						
Beta-BHC								
Delta-BHC								
Gamma-BHC								
Heptachlor								
Aldrin								
Heptachlor epoxide								
Endosulfan I								
Dieldrin								
4,4'-DDE								
Endrin								
Endosulfan II								
4,4'-DDD								
Endosulfan sulfate								
4,4'-DDT								
Methoxychlor								
Endrin ketone								
Endrin aldehyde								
Alpha chlordane								
Gamma chlordane								
Aroclor-1016								
Aroclor-1221								
Aroclor-1232								
Aroclor-1242								
Aroclor-1248								
Aroclor-1254								
Aroclor-1260								
Toxaphene								

Affected samples:

PBLK W1
EQK30
EQK31
EQK32
EQK32MS
EQK32MSD
EQK33

Pest/PCB

Reviewer's Initial/Date AV 6-12-92

3/90 Rev

* These flags should be applied to the analytes on the Sample Data Sheets.

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Midwestern Operations

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(313) 344-1770
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Clayton
ENVIRONMENTAL
CONSULTANTS

SDG NARRATIVE

Laboratory Name: Clayton Environmental Consultants (CLAYTN)
Case No.: 19026
EPA Sample Nos.: EQK13-19, EQK30-34
SDG No.: EQK13
Contract No.: 68-D1-0087

Case Summary

Case 19026 was received on April 30, 1992, and consisted of 7 soil samples for volatile analysis only, 4 water samples for full organic analysis, and 1 water sample for volatile analysis only.

The VOA water pH's are as follows:

Sample:	pH	Sample:	pH
EQK30:	1.8	EQK33:	1.7
EQK31:	1.8	EQK34:	1.8
EQK32:	2.2		

The Hewlett Packard GC/MS data system Clayton uses has a combined NBS/WILEY library. The data system prints the mass spectra for all tentatively identified compounds (TIC) and the top three library matches. When no library matches are found, the data system prints "NO DATA BASE ENTRIES RETRIEVED."

Standards

The instrument was tuned to meet the abundance criteria for BFB and DFTPP before any standards, blanks, or samples were analyzed. Response factor criteria for volatile and semivolatile target compounds, and volatile and semivolatile surrogate compounds met the minimum RRF criteria and maximum %RSD criteria for initial and continuing calibration data.

Surrogate Recoveries

Surrogate recovery results are reported on Form II and can be found in the QC summary data package.

002 RD06-03-92

Matrix Spike and Matrix Spike Duplicate

Matrix spike and matrix spike duplicate recovery data are reported on Form III and can be found in the QC summary data package.

Blanks

The method blanks with corresponding samples are reported on the method blank summary Form IV and can be found in the QC summary data package.

I certify that this data package is in compliance with the terms and the conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data submitted on floppy diskette has been authorized by the Laboratory Manager or his designee, as verified by the following signature.

Todd J. Outhouse
Project Leader, CLP

Date

003
RD 06-03-92

DATA REPORTING QUALIFIERS
(page 1)

For reporting results to EPA, the following result qualifiers are used. Additional flags or footnotes explaining results are encouraged. However, the definition of each flag must be explicit.

VALUE-if the results is a value greater than or equal to the Contract Required Quantitation Limit (CRQL), report the value.

U - Indicates compound was analyzed for but not detected. The sample Quantitation Limit must be corrected for dilution and for percent moisture. For example, 10 U for phenol in water if thee sample final volume is the protocol-specified final volume. If a 1 to 10 dilution of extract is necessary, the reported limit is 100 U. For a soil sample, the value must also be adjusted for percent moisture. For example, if the sample had 24% moisture and a 1 to 10 dilution factor, the Sample Quantitation Limit for phenol (330 U) would be corrected to:

$$\frac{(330\ U) \times df}{D} \qquad \text{where } D = \frac{100 - \%\ \text{moisture}}{100}$$

and df = dilution factor

at 24% moisture, $D = \frac{100 - 24}{100} = 0.76$

$$\frac{(330\ U) \times 10}{.76} = 4300\ U$$ rounded to the appropriate number of significant figures

For soil samples subjected to GPC clean-up procedures, the extract must be concentrated to 0.5 ml, and the sensitivity of the analysis is not compromised by the cleanup procedures. Therefore, the CRQL values will apply to all samples, regardless of cleanup. However, if a sample extract cannot be concentrated to the protocol-specified volume, this fact be accounted for in reporting the Sample Quantitation Limit.

J - Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed, or when the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the sample quantitation limit bur greater than zero. For example, if the sample quantitation limit is 10 ug/L, but a concentration of 3 ug/L is calculated, report it is as 3J. The Sample Quantitation Limit must be adjusted for dilution as discussed for the U flag. The J flag is also applied to pesticide/Aroclor results where the pesticide/Aroclor is confirmed to be present but the concentration is less than the CRQL.

N - Indicates presumptive evidence of a compound. This flag is only used for tentatively identified compounds. Where the identification is based on a mass spectral library search. It is applied to all TIC results.

DATA REPORTING QUALIFIERS
(page 2)

P - This flag is used for a pesticide/Aroclor target analyte when there is greater than 25% difference for detected concentrations between the two columns (see Form X). The lower of the two values is reported on Form I and flagged with a "P".

C - This flag applies to pesticide results where identification has been confirmed by GC/MS. If GC/MS confirmation was attempted but unsuccessful, do not apply this flag, instead use a laboratory-defined, discussed below.

B - This flag is used when the analyte is found in the associated blank as well as in the sample. It indicates possible/probable blank contamination and warns the data user to take appropriate action. This flag must be used for a TIC as well as for a positively identified TCL compound.

E - This flag identifies compounds whose concentrations exceed the calibration range of the GC/MS instrument for the specific analysis. This flag will not apply to pesticide/PCBs analyzed by GC/MS methods. If one or more compounds have a response greater than full scale, the sample or extract must be diluted and re-analyzed according to the specifications. All such compounds with a response greater than full scale should have the concentration flagged with an "E" on the Form I for the original analysis. If the dilution of the extract causes any compounds identified in the first analysis to be below the calibration range in the second analysis, then the results of both analyses shall be reported on separate Form I. The Form I for the diluted sample shall have the "DL" suffix appended to the sample number.

D - This flag identifies all compounds identified in an analysis at a secondary dilution factor. If a sample or extract is re-analyzed at a higher dilution factor, as in the "E" flag above, the "DL" suffix is appended to the sample number on the Form I for the diluted sample and all concentration values reported on that Form I are flagged with the "D" flag. This flag alerts data users that any discrepancies between the concentrations reported may be due to dilution of the sample or extract.

A - This flag indicates that a TIC is a suspected aldol-condensation product.

X - Other specific flags and footnotes may be required to properly define the results. If used, they must be fully described and such description attached to the Sample Data Summary Package and the SDG Narrative. If more than one flag is required, use "Y" and "Z", as needed. If more than five qualifiers are required for a sample result, use the "X" flag to combine several flags, as needed. For instance, the "X" flag might combine the "A", "B" and "D" flags for some sample. The laboratory-defined are limited to letters "X", "Y" and "Z".